
FOODS AND DRUGS

LABORATORY

OF THE

DEPARTMENT OF TRADE AND COMMERCE

OTTAWA, CANADA

BULLETIN No. 418

COCOA

NOTES AND COMMENTS.

Under this heading, as occasion arises, the Bulletins issued by this Department will contain, as an appendix, such comment as may seem necessary or advisable upon matters relating to the work of the Department in connection with the administration of the Adulteration Act, the Fertilizers Act, the Feeding Stuffs Act or the Proprietary Medicines Act.

It frequently happens that correspondents ask information regarding the above Acts, of such a nature that the matter in question possesses general interest, and comment upon it would prove acceptable and useful to others than the immediate questioner. In such cases the reply may find a place in these columns. For convenience of reference these notes will be numbered in series.

A. MCGILL,
Chief Analyst.

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OTTAWA, December 23, 1918.

F. C. T. O'HARA, Esq.,
Deputy Minister of Trade & Commerce,

Sir.—I beg to hand you a report upon the examination of 79 samples of Cocoa, purchased throughout Canada during the current year. We have on two former occasions done some work upon this article. The character and amount of this work is reported in Bulletin No. 79 (published September 1900) and in Bulletin 243 (published in October, 1912). The reports referred to showed that Cocoa and Chocolate were on the whole very desirable food articles and in the main free from anything that could be described as adulteration.

During the last four or five years the consumption of Cocoa in Canada has very greatly increased and quite naturally public interest in the origin, nature and variations of this article has also increased.

I placed this work in the hands of Mr. Collier of this staff and his report accompanying this is so carefully written and withal so comprehensive that I am sure it will be read with appreciation by cocoa users. Further than this the data he has collected together with that contained in previously mentioned bulletins will form a reliable basis for the enactment of standards for Cocoa and its preparations. We have not as yet established such standards for Canada.

Consumers of the article should be informed with regard to such matters as the quality of the article itself when genuine; whether or not it has been treated with alkali; whether or not added starch is present; whether or not excess of the cocoa husks is retained or added.

Unlike tea and coffee whose nutritional value as ordinarily used is negligible, Cocoa as a beverage is an article whose food value must be taken into account.

Owing to the absence of legal standards for this article, I have not classified the samples herein reported as genuine or otherwise. The matter of standardization will receive attention without avoidable delay.

I beg to recommend publication of this report as Bulletin No. 418.

I have the honour to be, Sir,

Your obedient servant,

A. MCGILL,
Chief Analyst.

Dr. A. MCGILL,
Chief Analyst,
Ottawa, Ont.

DEAR SIR.—I beg to submit herewith my report on the examination of a collection of 69 samples of cocoa. In addition 10 samples were analyzed by Mr. Davidson of the Winnipeg laboratory.

The demand for Cocoa products is rapidly increasing in all portions of the civilized world. As an example of the increased consumption of those products it may be noted that the importation of raw Cocoa beans into Canada has increased from less than three million pounds in the year 1910 to over six million pounds in the year 1916.

SOURCE AND MANUFACTURE.

Cocoa and Chocolate are products of the "beans" or seeds of several small trees natives of tropical America, of which *Theobroma cacao L.* is by far the most important. The value of cacao beans was known to the aborigines, especially the Aztecs of Mexico. The beans were not, however, extensively imported into Europe until the seventeenth century. At present the world's supply is chiefly derived from Brazil, Ecuador, and Venezuela in South America, Trinidad and other islands in the West Indies, San Thome in Africa, and Java and Ceylon in Asia.

The beans grow in large fruit or pods which are directly attached to the trunk or old wood of the tree. Each pod contains from 20 to 60 almond shaped seeds embedded in a mucilaginous pulp.

The gathered pods are first cut open, and the seeds with more or less adhering pulp are removed to boxes to undergo a process of "sweating" or fermentation. Besides loosening the adhering pulp, the enzymic action which is set up during this process produces certain chemical changes in the bean upon which its colour and flavour are largely dependent. After fermentation is complete, the beans are washed and dried. In some countries it is customary to rub the beans with dry earth to polish them before shipment.

On arrival at the factory, the beans are cleaned and sorted preparatory to roasting, which is then carried out in heated rotary drums. This important process develops the characteristic aroma and colour of the bean, removes certain astringent constituents and dries the bean so that the so-called "shell" which surrounds the kernel of the seed is rendered brittle and non-adherent.

After cooling, the roasted beans are passed through "nibbing" machines which crack them in order to permit of the kernels being freed from the shell and "germ" by winnowing and sifting.

The cracked kernels, known as "cacao nibs", are next ground to a fine paste in steam heated mills.

If chocolate is to be prepared this paste or "cacao mass" is usually mixed and ground with sugar and spices, after which it is finished by appropriate methods.

In the preparation of cocoa, however, it is customary to remove about half the fat contained in the ground nibs by expression in steam heated hydraulic presses. The pressed mass thus obtained is then finely ground and sifted to form the cocoa of commerce. It is held that the removal of the excess of fat tends to improve the cocoa from a dietetic standpoint.

COMPOSITION AND NUTRITIVE VALUE OF CACAO PRODUCTS.

Cacao beans from different sources show considerable variation in composition. This is accounted for by differences of botanical origin, climate, soil, or methods of preparation. The following analyses will indicate the typical composition of some of the leading cacao products:—

Determination.	Roasted Cacao Nibs.	Roasted Cacao Shells.	Cocoa Powder.	Sweet Choco- late.
Moisture.....	2·72%	4·87%	3·99%	2·15%
Fat.....	50·12%	37·77%	25·27%	25·53%
Total ash (Mineral Matter).....	3·22%	10·48%	4·97%	1·72%
Theobromine.....	1·04%	0·49%	1·56%	0·68%
Caffeine.....	0·40%	0·10%	0·60%
Other Nitrogenous Substances (Proteins, etc.).....	12·12%	14·54%	18·18%	6·95%
Crude Fibre (Cellulose).....	2·64%	16·63%	3·96%	1·40%
Starch (By Diastase Method).....	8·07%	4·14%	12·11%	5·46%
Other Nitrogen Free Substances.....	19·57%	46·40%	29·36%	9·54%
Sucrose.....	46·58%

The figures in the first two columns are from analyses by Winton, Silverman and Bailey. The results in the other columns are compiled from various sources.

Fat.—The fat present in cocoa consists of cacao butter oil of *Theobroma*. This is a white edible solid melting at 33°C. The fat has a bland agreeable taste, and is characterized by the fact that it seldom becomes rancid.

Theobromine is the principle alkaloid of cocoa though small quantities of caffeine are also present. Cocoa owes its slightly stimulating properties to the presence of these alkaloids, but the physiological action of theobromine is not as marked as that of caffeine, the alkaloid present in coffee. None of the symptoms ascribed to the use of tea or coffee have been noted as resulting from the employment of cocoa as a beverage. (Davis: Food in Health and Disease, 2nd Edn., p. 134.)

The nitrogenous constituents of cocoa other than the alkaloids have been but little studied. Dekker, however, has shown (Chem. Centr., 2, 1217) that they consist largely of proteins.

Starch.—The amounts of starch and other carbohydrates naturally present in cocoa vary considerably. The starch and dextrine besides possessing considerable nutritive value help to hold the insoluble portion of the cocoa in suspension when it is mixed with boiling water.

In addition to the constituents considered above, small quantities of the salts of organic acids, and the colouring matter cacao red are also present.

Cocoa as a Beverage.—The method of preparing cocoa for use as a beverage differs materially from that employed in making tea and coffee. In the case of the latter beverages, only the constituents which have been dissolved by the boiling water are added to the cup. When cocoa is employed, all the vegetable tissue forms a part of the beverage. About 21 per cent of the finely ground solid is soluble in boiling water, the remainder being merely held in suspension by the liquid.

It will be apparent that the nutritive value of cocoa as a beverage is considerable. On the other hand tea and coffee cannot be regarded as foods apart from the milk and sugar usually consumed with them. As a result of determinations on two samples of cocoa made in this laboratory by means of the calorimeter, the following calorific values were obtained:—

(1) 5.60 large calories per gram, 158.8 calories per ounce or 2541 calories per pound.

(2) 79.2 large calories per cup.

Sample 1 was a cocoa powder of average composition. Sample 2 was a mixture of this cocoa with milk powder and sugar in the amounts that are usually present in one cup of the beverage (200cc), namely, cocoa, 1 teaspoon (4 grams); sugar, 1 teaspoon (5 grams); milk powder (fat-free), 10 grams, equivalent to one-half cup of milk.

Cocoa Shell.—It has been previously noted that the husk or shell of the cocoa bean is removed from the kernel during the course of manufacture. The shell constitutes from 8 to 15 per cent of the roasted bean, though in some cases it may amount to as much as 20 per cent.

The profitable utilization of the shell has occupied the attention of numerous chemists and agriculturists, but much still remains to be done before the problem can be considered solved. Among the uses proposed for the husk may be mentioned its employment as a source of fat and theobromine, a feeding stuff for cattle, and as a manure. (*Jour. Soc. Chem. Ind.*, July 31, p. 240T).

For many years the poorer classes of certain countries of Europe have employed an infusion of the shells as a beverage under the name of "miserables" or "Cocoa Tea." While not unpalatable, this infusion has little nutritive value. Recently an attempt was made to popularize the use of the husk as a beverage in Great Britain by placing it on the market under fancy names at an exorbitant price. (*Analyst*, June, 1918, p. 192.) There seems to be no objection in Great Britain to the sale of the shell for use as a beverage provided it is sold at a reasonable price. The British Cacao Powder Order, 1918, forbids the sale of shell or mixtures containing more than 5 per cent shell (except chocolate) at a retail price exceeding 6d. per pound.

SOLUBLE COCOAS.

The term "soluble" is generally applied by the manufacturer to cocoas which have been subjected to the "alkali treatment." From the fact that this method of treatment originated in Holland it is often known as the 'Dutch process.'

The removal of a portion of the fat in the course of manufacture renders the cocoa powder considerably lighter in colour. For years it has been the endeavour of manufacturers to darken the colour of the powder so that it would more closely resemble the rich hue of chocolate. This effect has been sought in many ways but the most prevalent method is by the addition of alkalies or alkaline carbonates. Ammonium carbonate, and magnesium carbonate, have also been employed with this end in view. Attempts have been made to develop this enhanced colour by other means than by the addition of chemicals such as by treatment with steam under pressure, etc.

While its effect upon the colour of the cocoa was probably the original cause of the employment of the alkali treatment, it was subsequently claimed that it renders the powder more soluble in water.

The treatment is carried out through the agency of small quantities of one or more of the following reagents: Potassium hydroxide, potassium carbonate, potassium bicarbonate, sodium hydroxide, sodium carbonate, sodium bicarbonate, magnesium carbonate, ammonia, and ammonium carbonate. The methods used in applying these chemicals vary considerably, but the salts are usually added to the nibs or cocoa in the form of a weak aqueous solution, or in the case of magnesium carbonate as an emulsion with water. Heat is then applied until the water is driven off. Ammonia is sometimes used in the gaseous form. When ammonia or ammonium carbonate is employed, the treatment should take place previous to roasting as a high temperature is required to remove the odour of ammonia from the cocoa.

While it is generally agreed that the alkali treatment produces a cocoa which forms a more perfect suspension in the cup, the claim that this treatment renders the cocoa more soluble in water has been known for some years to be false. The conclusions arrived at as the result of an exhaustive investigation of this claim undertaken recently by the United States Bureau of Chemistry (United States Department of Agriculture, Bulletin 666) may be briefly summarized as follows:—

- (1) The chief effect of the alkali treatment is apparently the increase in colour of the water solution.

(2) The total water-soluble matter in treated cocoas is slightly increased, but this is more than accounted for by the added alkali present.

(3) There is a slight increase in the amount of water-soluble proteins by the alkali treatment. The amount of the non-nitrogenous substances soluble in water is on the contrary decreased by this treatment.

(4) The alkali treatment increases the ratio of the soluble ash to the insoluble ash. This holds good for all the alkalies employed, except ammonium carbonate and magnesium carbonate. Both of these, however, increase this ratio in the ash of the water-soluble matter.

This investigation has shown conclusively that the claim that the alkali treatment increases the amount of cocoa soluble in water is without foundation.

The United States Board of Food and Drug Inspectors have stated their attitude towards the use of the word "soluble" in the following decision (United States Department of Agriculture, Food Inspection Decision 180): "In the opinion of the board, cocoa not treated with alkali is not soluble in the ordinary acceptation of the term. Cocoa before and after treatment with alkali shows essentially the same lack of solubility. To designate the alkali-treated cocoa as "soluble" cocoa is misleading and deceptive."

Authorities differ as to the advisability of the alkali treatment of cocoa regarded from a hygienic standpoint. The opinions of certain of them are summed up by Whymper (Cocoa and Chocolate, p. 104) as follows: "The addition of alkalies to cocoa has not met with unqualified approval, and there are many who, while readily acknowledging the superiority of the beverage prepared from beans or cocoa so treated, are not prepared to admit that the inclusion of chemicals is not prejudicial to health."

In considering the desirability of the addition of alkalies from the point of view of health, and the need of regulating the quantity added by law, Bordas shows that the natural cacao mass contains from 2.46 per cent to 3.05 per cent of potash estimated as K₂O on the dry fat-free basis, whilst samples of "soluble" cocoas contain from 4.82 per cent to 6.41 per cent of the same material. He points out that while some countries restrict the amount of alkali that may be added, others forbid its use entirely. The same author concludes that, as there is no direct evidence of injury to health by the consumption of treated cocoas, there is no harm in the addition of alkalies provided that the quality is not permitted to be excessive (Ann. Falsific. 1910, 3, 61-70).

STANDARDS FOR COCOA.

Though the provisions of the *Food Adulteration Act* of Canada apply to cocoa, no special standards are in force in this country as yet.

The following standards were established by the United States Government in 1906:—

Cocoa beans are the seeds of the cacao tree, *Theobroma cacao* L.

Cocoa nibs, cracked cocoa, is the roasted broken cocoa bean freed from its shell or husk.

Chocolate, plain chocolate, bitter chocolate, chocolate liquor, bitter chocolate coatings, is the solid plastic mass obtained by grinding cocoa nibs without the removal of fat or other constituents except the germ, and contains not more than three (3) per cent of ash, three and fifty hundredths (3.50) per cent of crude fibre, and nine (9) per cent of starch, and not less than forty-five (45) per cent of cocoa fat.

Cocoa, powdered cocoa, is the cocoa nibs, with or without the germ, deprived of a portion of its fat and finely pulverized, and contains percentages of ash, crude fibre, and starch corresponding to those in chocolate after correction for fat removed.

Sweet cocoa, sweetened cocoa, is cocoa mixed with sugar (sucrose) and contains not more than sixty (60) per cent of sugar (sucrose) and in the sugar and fat-free residue no higher percentage of either ash, crude fibre, or starch than is found in the sugar and fat-free residue of chocolate.

It is also provided by United States Food Inspection Decision 136 that 'when a cocoa is treated with an alkali or an alkaline salt, as in the so-called Dutch process, and the finished cocoa contains increased mineral matter as the result of this treatment, but no alkali as such is present, the label should bear a statement to the effect that the cocoa contains added mineral ingredients, stating the amount. Cocoas and chocolates containing an appreciable amount of free alkali are adulterated.'

Until the present year, there were no standards for cocoa in force in the United Kingdom. This year the British Food Controller promulgated the Cocoa Powder Order, 1918. This order specifies two grades of cocoa powder, "Grade A" and "Grade B". Part I of this order declares that after April 8, 1918, no cocoa powder shall be manufactured which contains more than 5 per cent of cacao bean shell, and which contains not less than 22½ per cent and not more than 30 per cent cacao butter. Such powder must be invoiced and labelled as "Grade B Cocoa Powder." Clause 3 provides that a manufacturer may sell packed in cartons as "Grade A Cocoa Powder" any cocoa powder which contains not more than 2 per cent cacao bean shell. This order also fixes maximum retail prices for the above products as well as for cacao shell.

Adulteration of Cocoa.—The necessity for fixing standards for cocoa is evident from a consideration of the adulterations to which it is subject. The most commonly employed adulterants are starches, cacao shell and sugar. The starches used are generally those of arrowroot, sago, and maize. To a lesser extent, ground wood fibre and mineral matter, such as iron oxide, are added to cocoa. The use of the latter adulterants, however, is rare at the present time. Starch is sometimes added for the alleged purpose of diluting the cocoa fat instead of removing a portion of it by pressure. It is claimed that the dilution of the cocoa by this means renders it more digestible and nutritious.

It seems plain that, whatever may be the reason for the addition of starch or other ingredients to cocoa, it constitutes adulteration unless such addition is stated so distinctly on the label of the package that the consumer is made aware of what he is getting.

DISCUSSION OF ANALYTICAL DATA.

The samples of cocoa in this collection as purchased by the Department's inspectors may be classified as follows:—

Collected in original containers, 64 samples.

Collected in bulk, 15 samples.

The former comprise twenty-six different brands. Of the latter, six samples were of unknown origin. The samples purchased were designated as cocoa, pure cocoa, soluble cocoa, etc. The manufacturers of four of these brands acknowledge more or less definitely the addition of substances other than cocoa, but only two of these bear labels specifying the composition of these additions. They are:—

No. 79752—Tibbles Vi-Cocoa, "prepared from cocoa, kola, malt and hops."

No. 70467—"Pure Gold Instantaneous Milk cocoa (sweetened)."

The analytical work on the collection was undertaken in order to answer the following questions:—

(1) What is the general character of the cocoas on the Canadian market?

(2) How many of these cocoas give evidence of having been subjected to the "alkali treatment," and what is the amount of alkali present in such?

(3) To what extent have the cocoas in this collection been adulterated?

In order to furnish data from which an answer to these questions could be formulated, it was considered necessary to make the determinations enumerated below:—

(Moisture (loss at 105°C.).

(2) Fat (Petrolic Ether Extract).

(3) Ash (a) total ash, (b) water-insoluble ash, (c) water-soluble ash, (d) percentage of total ash soluble in water, (e) alkalinity of total ash.

(4) Crude fibre (moisture-fat-free basis).

(5) Microscopical examination.

(6) Sugar (sucrose).

(7) Milk solids, including casein and lactose.

(8) Starch (by acid hydrolysis).

Moisture.—The moisture content of the samples varied from a maximum of 7.20 per cent (81267) to a minimum of 1.68 per cent (84130). These figures must, however, be regarded as abnormal as 90 per cent of the samples contain from 3 to 6 per cent of moisture. An excess of moisture tends to "ball" the powder and render it liable to the attack of mildew.

Fat.—This was estimated by continuous extraction for 16 hours with petrolic ether. The proportion of fat present in the eighty samples examined may be classified as follows:—

30-32 per cent fat, four samples.

20-30 per cent fat, sixty samples.

11-20 per cent fat, ten samples.

5-11 per cent fat, five samples.

It will therefore be evident that over 81 per cent of the samples contain more than 20 per cent of fat. All of the samples which contain less than 11 per cent and more than half of those containing between 11 and 20 per cent fat, have been found to include other substances besides cocoa. In conjunction with other determinations, the fat content is a guide in deciding as to the presence in cocoa of other substances such as starch, cocoa shell, etc.

Ash.—The ash determinations are valuable as indications, when cautiously interpreted, of the addition of mineral and other foreign substances to cocoa. Thus in the case of the total ash, the treatment of the cocoa with alkalies (with the exception of ammonium salts) would cause the ash to be increased. On the other hand, the addition of substances such as starch and sugar will decrease the ash below that of an unadulterated cocoa. The water-soluble ash will, of course, be increased by the use of a soluble alkali in treating the cocoa, and is therefore of value in determining whether such treatment has been given.

It has been pointed out by many authorities on the subject that the percentage of the total ash that is soluble in water in an untreated cocoa is invariably less than 50 per cent. On the other hand, when a cocoa has been treated with the salts of sodium and potassium, the alkalies most generally employed, the percentage of total ash soluble in water will be greater than 50 (usually from 60 per cent to 80 per cent). Ammonium salts, being volatile, have no effect upon the amount of the ash. Owing to its insolubility magnesium carbonate increases the percentage of the water insoluble ash and at the same time the presence of this salt is indicated by an increased alkalinity of the total ash. From the foregoing it will be evident that when the figures in the column headed "Percentage of Total Ash Soluble in Water" in the accompanying tables are greater than 50 per cent it can be said with certainty that samples showing such percentages have been treated with alkalies, though, for the reasons stated above, it does not necessarily follow that all samples in which this percentage is less than 50 are untreated.

Alkalinity of Total Ash was determined by adding excess of tenth normal acid to the total ash and titrating back to neutrality, using methyl orange as an indicator. The results obtained are expressed in terms of cc of N/10 acid required to neutralize the total ash from one gram of moisture-fat-free cocoa. In the literature published on this subject the term "alkalinity of ash" is often used so ambiguously that it is difficult to determine how the results given under this title were arrived at. On this continent it appears to be customary to state the alkalinity in terms cc. of acid required per gram. In Europe the amount of acid used is calculated to either K₂O or K₂O₃.

TABLE I.—Cocoas manufactured in Canada or the United States.
Averages of samples of each brand analyzed.

Sample No.	Manufacturer.	Moisture.	Fat.	Total Ash.	Water Insol. Ash.	Water Soluble Ash.	Percentage of Total Ash Soluble in water.	Alk. of Total Ashcc. of N/10 Acid.
On Moisture-Fat-Free basis.								
7691, 81269, 83421, 84124, 84408, 79744...	Baker's.....	3·60	25·21	7·74	5·29	2·48	32·2	8·46
7685, 70463, 81256, 83412, 84405, 79745...	Cowan's.....	3·96	25·07	7·39	4·32	2·57	35·1	7·96
73746, 80009, 81268...	Fry's, U.S.A....	3·98	24·48	8·28	2·88	5·40	65·3	10·33
79749, 70462, 7687, 80003, 83426...	Lowney's.....	4·15	24·03	7·11	4·43	2·68	37·7	7·92
80008.....	Barbours Acorn..	5·80	23·20	8·88	2·11	6·77	76·2	10·98
79747.....	Hershey's.....	3·55	22·75	7·19	4·42	2·77	39·0	5·70
80572, 80633...	Todhunter's.....	3·81	27·20	6·26	4·24	2·02	32·2	7·22
80005, 83425...	Mott's.....	3·90	30·37	6·69	3·77	3·42	44·1	8·40
70464.....	Litster's.....	4·78	20·65	7·53	4·56	2·97	39·5	8·71
81273.....	Dalton's.....	4·54	30·15	7·93	4·50	3·43	43·2	8·57
80571.....	Lowe's.....	4·50	20·90	7·69	4·45	3·24	42·2	9·31
83424.....	Schwartz's.....	5·72	25·20	9·42	2·06	7·36	77·7	13·24
79751.....	White Star.....	5·70	20·75	9·86	2·25	7·61	77·0	8·90
80006.....	Litster's Ever Ready.....	2·98	5·42	3·06	2·09	0·97	31·4	3·16
70467.....	Pure Gold Milk Cocoa	3·70	5·25	4·83	2·92	1·91	39·5	5·16

Samples Nos. 80006 and 70467 are mixtures of cocoa with milk powder and sugar, and therefore give results differing considerably from those given by straight cocoa. The presence of these ingredients is declared on the label of sample No. 70467, but not on that of the other sample. The total ash of the samples listed above varies between a maximum of 9·86 per cent (79751) and a minimum of 3·06 per cent (80006). Four of the brands, viz., Fry's, "Acorn," Schwartz, White Star, give a total ash of 8 per cent. The high percentage of the water-soluble ash indicates that the samples of these brands have been subjected to the alkali treatment. This treatment is possibly intended to be acknowledged by the use of the word "Dutch" on the labels of the two latter brands. There is no indication that the claims for purity and freedom from chemicals made for the other brands listed in the above table are not correct.

TABLE II.—Cocoas of European Manufacture.

Averages of samples of each brand analyzed.

Sample No.	Manufacturer.	Moisture.	Fat.	Total Ash.	Water Insoluble Ash.	Water Soluble Ash.	Percentage of Total Ash Soluble In Water.	Alk. of T. A. per gm.	N/10 Acid.
On Moisture-Fat-Free basis.									
79743, 7680, 79986, 84409	Fry's, England....	5.64	22.40	8.97	1.96	7.02	79.1	12.96	
80002, 80675, 31276, 83423, 84121,	Bensdorp's.....	5.45	29.45	10.06	2.13	7.93	78.8	12.58	
79750	Cadbury's.....	5.30	22.65	10.76	2.09	8.67	81.0	12.28	
80711	De Jong.....	5.43	28.05	11.83	1.79	10.04	84.9	15.33	
79748, 79987, 81267, 80625,	Epp's Pure Un-sweetened	6.06	15.44	8.29	4.36	8.12	51.2	9.47	
70466, 79985, 80007, 80609, 81270,	Lipton's.....	5.00	23.13	8.30	2.43	5.97	71.8	11.68	
70458, 80689, 84106	Suchards Soluble Coccos	4.41	29.73	9.95	2.29	7.66	76.7	12.40	
79752	Tibbles Vi-Cocoa.....	4.55	23.95	10.14	2.31	7.83	77.0	9.51	
84123	Tobler's.....	5.46	28.00	10.21	1.95	8.26	80.9	13.20	
7679, 70459, 80684, 84410	Fry's Homeopathic.....	4.07	15.76	2.46	1.68	0.78	31.5	2.74	
70461, 80712,	Van Houten's.....	4.41	25.73	11.85	1.88	9.97	84.1	14.67	

All of these brands of cocoa, with the exception of Fry's Homeopathic, give decided evidence of the addition of alkalies in their manufacture. With this exception, the average total ash of these samples varies between 8.29 per cent and 11.85 per cent. Of these, the water-soluble ash amounts to from 51.2 to 84.9 per cent of the total ash. Most of the samples of the same brand when analysed showed commendable uniformity of composition. In the case of Epps, however, the different samples of apparently the same brand seemed to vary more than usually is the case.

Apart from the use of such terms as "soluble," "solubilised," etc., none of these brands bear labels declaring that alkalies have been added. Bensdorp's cocoa, however, is labelled "prepared by a special process whereby the mineral ingredients of this cocoa have been increased by 3 per cent."

The low figures obtained from the determinations of ash, fat, and alkalinity on the samples of Fry's Homeopathic cocoa are due to the presence of added starch in the cocoa. A direct determination of starch in conjunction with a microscopical examination indicates that approximately 33 per cent of arrowroot has been added. The label of this brand of cocoa bears the following ambiguous statement:—

"Manufactured with cocoa carefully selected for quality and fine flavour and other ingredients, the perfect purity and wholesomeness of which are guaranteed." The wholesomeness of arrowroot is beyond question, but unless its presence is specifically declared on the label of the package the public will not know that the contents consist of a mixture of cocoa and arrowroot.

In the following table I have summarized the figures for the ash tabulated in the two preceding ones. This table has been subdivided according as to whether the water-soluble ash of the brands of cocoa amounts to more than or less than 50 per cent of the total ash. Those with more than 50 per cent have been treated with alkali. Samples which contain ingredients other than cocoa have been excluded in compiling this table. The alkalinity is expressed as in the preceding tables and has also been calculated as is customary in Europe to potassium oxide and potassium carbonate.

TABLE III.—Mean Results on Cocoas classified according to percentage of water-soluble ash.

Determination.	(9 Brands.)			(13 Brands.)			Untreated Cocoa. (Bull. 666, U.S.A.)	
	Water-Soluble Ash Less than 50% of Total Ash.			W.—Soluble Ash Greater than 50% of Total Ash.				
	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.		
Total ash p.c.	7.93	6.26	7.28	11.85	8.29	9.11	7.62	
Insoluble ash "	5.29	4.24	4.50	4.36	1.79	2.31	5.06	
W.-soluble ash "	3.43	3.02	3.84	10.04	5.13	7.59	2.54	
Alkalinity Cc.	9.31	7.22	8.03	15.33	8.90	12.16	7.45	
" K ₂ O, p.c.	4.37	3.3	3.77	7.20	4.18	5.71	3.51	
" K ₂ CO ₃ p.c.	6.42	4.98	5.54	10.58	6.14	8.39	5.14	

The figures quoted in the last column are the result of analyses of a number of authentic samples of untreated cocoa. (United States Department of Agriculture Bulletin 666). By comparison with the figures in the preceding column they will serve as an indication of the amount of alkali that has been added to the treated samples. In this connection it may be noted that the French official decree of December 17, 1910, regarding alkalis cocoas, provides that the quantity of added alkali should not exceed 5.57 gms. of potassium carbonate or an equivalent of other alkaline carbonate per 100 gms. of dry fat-free cocoa, and also requires that the powder should retain a faintly acid reaction without the addition of acidifying substances.

Microscopical Examination.—This examination provides a most valuable means of determining whether foreign substances have been added to the cocoa. During the work on this collection at least one sample of each brand of the packaged cocoas was examined microscopically. Where the samples had been purchased in bulk they were all examined.

Each sample was first examined in a water mount for the detection and identification of the presence of foreign starches, etc. Nine samples were thus found to contain other starch besides the natural cocoa starch. Comparison was made with pure cocoa and with mounts of the different starches.

Subsequently a mount was made from each sample in a chloral hydrate medium for the identification of the cocoa nib tissue and the detection of the presence of cocoa shell.

The addition or retention of cocoa shell during manufacture constitutes one of the most common forms of adulteration of cocoa and chocolate. The methods employed by analysts for the detection and estimation of shell in cocoa and chocolate may be classified as mechanical methods such as the levigation process; chemical methods for the determination of crude fibre, pentosans, nitrogen, etc.; and microscopic methods by which the vegetable cells peculiar to the husk may be detected. Unfortunately all of these methods give results which have not attained the accuracy desirable. In connection with the recent British Cocoa Order it was pointed out to the Food Controller by the Society of Public Analysts that the satisfactory differentiation between 2 and 5 per cent of cocoa shell in cocoa powder was in most cases impossible. (Analyst June, 1918, p. 201.) The difficulties encountered in the determination of added shell are increased by the fact that, in spite of the efficient methods employed by the honest manufacturer to remove the husk from the nib, a small proportion of the former still remains. Knapp (Jour. Soc. Chem. Ind., vol. 37, p. 241-T) states that continuous vigilance is required to prevent the proportion of shell in the nib from exceeding 2

per cent. Beythien and Pannwitz state (Chem. Abs. vol. XI, p. 1214) that commercial cocoa almost always contains some shell tissue. As the result of work reported from various sources the maximum tolerance for shells in commercial cocoa is placed at 4.4 per cent (moisture and fat-free basis).

It will be evident from a consideration of the above, that the microscopical estimation of added cocoa shell resolves itself into a determination as to whether shell is present in excess of the amount retained during careful manufacture under commercial conditions. In the absence of definite stipulations to the contrary, it would appear that no cocoa which contained less than 5 per cent of shell could be considered adulterated with excessive shell. Microscopically, cocoa shell is characterized by the presence of stone cells, mucilage cells, and large spiral vessels. By counting the number of stone cell groups in each mount and comparing this with mixtures of shell and nib of known composition it is possible to ascertain how much shell a sample of cocoa contains. After preparing standard samples of pure shell and nib from the original beans the samples included in this collection were examined in the manner specified. As the result of this examination I have come to the conclusion that although all of the samples contain small proportions of shell, in none of them does the amount exceed 5 per cent. In reporting my results I considered it was unnecessary in view of the small amount present to mention the presence of shell specifically.

Crude Fibre.—This determination was made according to the official method. It forms perhaps the most reliable chemical means of detecting the presence of added shell in cocoa, and the findings from this determination are especially valuable when interrupted in conjunction with the results of the microscopical examination or levigation method. Beythien and Pannwitz give the following values for crude fibre:—

Cocoa nibs (moisture-fat-free basis) 5.69—8.86 per cent.

Cocoa shell (moisture-fat-free-basis) 11.84—21.17 per cent.

The same authors state that a crude fibre of 2.5—9.0 per cent (moisture-fat-free-basis) is suspicious, the strength of evidence being increased in degree as the amount exceeds 9 per cent.

The crude fibre was determined on each of the samples collected in bulk, and upon at least one sample of each of the brands in packages. The maximum percentage of crude fibre was 8.26 per cent (79987), the minimum was 1.17 per cent (80684). This low figure was due to the presence of foreign starch in the cocoa. It will be noted that the maximum figure is below the limit prescribed by the authorities quoted above.

The methods employed in estimating Sucrose, Lactose, and Casein, do not call for any special comment. The latter determinations were undertaken for the purpose of estimating the proportions of milk powder present in two of the samples. The results of the three determinations were calculated as percentages of the original samples.

Starch.—A determination of the total starch was made (on moisture-fat-free basis) by acid hydrolysis on samples in which the addition of foreign starches had been detected as a result of the microscopical examination. By deducting from the total starch the average (23.66 per cent starch by acid hydrolysis method, (Leach, "Analysis of Foods," p. 395) amount found in pure cocoa nibs, it was possible to arrive at an approximate estimate of the starch added.

The following table will indicate the amount of starch and other constituents present in the samples purchased in bulk:—

TABLE IV.—Composition of samples purchased in bulk.

Sample No.	Manufacturer.	Moisture.	Fat.	Total Ash.	W.-Insol. Ash.	W.-Sol. Ash.	Crude Fibre.	Added Starch. (Approx.)	Sucrose.
70465	J. S. Fry	5.96	6.45	1.48	0.94	0.54	1.46	68.2
80010	W. Baker	3.80	23.00	7.70	4.53	3.17	4.82
80068	Unknown	3.58	25.80	6.99	3.25	3.14	5.65
80066	J. P. Mott	2.64	11.25	4.03	3.48	1.15	4.76	2.6	39.6
81271	Todhunter M. Co.	4.06	23.55	7.10	4.41	2.08	5.49
84125	W. Baker	4.34	26.35	7.88	6.08	1.80	4.19
84126	Unknown	3.36	26.30	7.73	4.86	2.88	6.18
84127	Todhunter M. Co.	3.64	15.20	4.31	3.54	1.77	3.30	26.1
84128	"	3.54	18.95	3.83	2.05	1.77	2.42	26.2
84129	Cowan	2.86	9.80	2.77	1.81	0.98	3.25	8.1	42
84130	Unknown	1.68	7.25	2.59	0.28	1.66	52.3
84404	J. Cowan	3.48	26.30	7.68	4.86	2.82	6.36
84411	Unknown	4.30	21.35	7.67	4.57	3.10
84414	"	4.00	20.90	6.60	4.07	2.53	5.58
84415	"	4.20	25.80	7.03	4.48	2.54	5.44

It will be noted that six of these samples contain added starch or sugar. The names of the manufacturers are those furnished to the inspector by the vendor. A comparison between the cocoa supplied by some of these manufacturers in packages with that supplied in bulk is striking. The former is of good quality, but the bulk cocoa said to be of their manufacture contains so much starch or sugar that the proportion of cocoa matter is considerably reduced.

A study of the data supplied in this report indicates that the majority of the manufacturers devote considerable care to the maintenance of the purity and uniformity of their product. It also suggests that the time is ripe for this country to follow the lead of the United States and other countries in establishing standards for cocoa. If properly drawn up and enforced it is probable that such standards would be welcomed alike by the consumer and the manufacturer. Their establishment would tend to increase the confidence of the Canadian public in the quality of the cocoa on the market and would render unnecessary such statements as "It has more than three times the strength of ordinary cocoa containing starch." The Canadian manufacturer who maintains the purity of his product would seem to be entitled to protection from the competition of his less scrupulous rival.

SUMMARY.

- (1) The sources of the cocoa bean are indicated, and its manufacture into cocoa of commerce is described.
- (2) The composition and nutritive value of cocoa products are considered.
- (3) The effect of the employment of alkalies in the preparation of so-called "soluble" cocoas is discussed.
- (4) The standards for cocoa in force in the United States and the United Kingdom are reviewed, and the adulteration to which this product is subject are pointed out.
- (5) The reasons for making this collection of cocoas, and the determinations necessary to form a judgment of their quality are indicated.
- (6) The significance of the fat and ash determinations in the detection of adulteration are pointed out.

(7) Tables are given showing the character of cocoas of European and American manufacture with special reference to the composition of the ash as an indication of the addition of alkalies.

(8) The detection of added cocoa shell by means of the microscopical examination supplemented by the determination of crude fibre is discussed.

(9) A table is given showing the composition of the samples purchased in bulk. It is shown that in some cases they are of inferior value as cocoa owing to the presence of starches and sugar.

(10) Thirteen of the samples examined have been found to contain other ingredients besides cocoa (not including the addition of alkalies). In only two cases were such additions acknowledged specifically. Of the twenty-six brands of cocoas obtained in package form, fourteen gave evidence of treatment with alkalies. Most of these were of European manufacture.

I have the honour to be, Sir,
Your obedient servant,

F. C. COLLIER,
Public Analyst.

BULLETIN No. 418—COCOA.

No. of Sample	Name of Manufacturer, etc.	Moisture, etc.	Fat.	Results of Analysis.							Remarks, and Opinion of the Chief Analyst.	
				On Moisture—Fat—Free Basis.				Other Ingredients.	Microscopical Examination.	Purchased.		
				Ash.	Per centality of total Ash	Alkalinity of total Ash c.c.s.	Crude Fibre.					
		%	%	%	%	%	%	%	%			
7679	Fry's Homeopathic	4.40	15.65	2.47	1.66	0.81	32.8	2.81	Cocoa tissues and arrowroot.	
7680	Fry's	6.74	22.06	8.84	1.93	6.91	78.2	13.66	6.43	
7685	Cowan's	3.65	26.70	7.33	4.54	2.79	38.1	8.72	7.35	
7687	Lowney's	4.04	25.30	7.16	4.13	3.03	42.3	8.84	
7691	Baker's	3.50	24.25	7.47	4.79	2.68	35.9	8.31	
7695	Suehard's Soluble	4.34	29.15	10.41	2.15	8.26	79.4	12.93	6.62	
7696	Fry's Homeopathic	3.78	15.93	2.39	1.69	0.70	29.2	2.61	
7697	Van Houten's	4.68	25.97	11.88	1.87	10.01	84.2	14.78	
7698	Lowney's	4.30	25.90	6.78	4.00	2.60	39.7	8.44	
7699	Cowan's	4.35	24.55	7.20	4.64	2.56	35.6	8.30	
70464	Lipton's	4.76	20.65	7.53	4.56	2.97	30.5	8.71	6.40	
70465	Fry's	5.86	6.45	1.48	0.94	0.54	36.4	1.86	11.46	Added starch, 68.2	Coconut tissues and arrowroot.	
70466	Lipton's Pure Gold Milk Cocon.	5.58	23.05	9.16	1.85	1.31	79.8	12.26	2.23	Source 31.2% milk solids, non fat, 22.75%.	Coconut tissues and arrowroot.	
70467	Pure Gold Milk	3.70	5.25	4.83	2.93	1.91	39.5	5.16	
70895	Lipton's	4.80	22.40	7.53	1.95	5.58	74.1	11.53	
70896	Fry's	5.66	21.65	8.96	1.82	1.14	79.7	13.63	
70897	Epsom Pure Un-sweetened.	6.88	13.35	8.93	8.86	5.06	86.7	10.96	8.26	Coconut tissues.	
80002	Benedorp's	5.30	26.70	9.61	2.27	7.34	76.4	12.05	5.19	
80003	Lowney's	4.86	23.30	4.33	2.76	8.35	39.0	8.35	5.77	Coconut tissues.	

BULLETIN No. 418—COCOA.

No. of Sample	Name of Manufacturer, etc.	Moisture.	Fat.	Results of Analysis.						Purchased.	Remarks, and Opinions of the Chief Analyst.		
				On Moisture—Fat-Free Basis.				Other Ingredients.	Microscopical Exam. sites.				
				Ash.	Water Soluble.	Water Insoluble.	Total.	Alka. Per centinity of total Ash Soluble C.C.A. N/10 "id. per gm.	Crude Fibre.				
84129	Cowans.....	2.86	9.80	2.77	1.81	0.96	34.7	7.21	3.25	Starch 8.1% sugar 42.6% Sugar 52.3%	Cocoa tissue and In bulk... maize starch.		
84130	Unknown.....	1.68	7.35	2.58	0.93	1.66	64.1	2.58	6.36	"	"		
84404	Cowan's.....	3.48	26.35	7.66	2.57	5.00	65.4	10.61	6.36	"	"		
84405	".....	3.85	26.05	8.20	5.99	2.21	27.0	8.56	"	"	"		
84406	Suchard's Soluble.....	5.02	28.25	10.47	2.22	8.25	78.8	12.99	"	"	"		
84408	Baker's.....	3.34	24.25	8.20	6.44	1.76	21.5	9.13	"	"	"		
84410	Fry's Homeopathic.....	5.30	24.25	9.12	2.10	7.02	77.0	13.65	2.73	"	Cocoa tissue and arrowroot.		
79743	Fry's.....	3.80	15.60	2.61	1.60	0.82	32.7	"	"	"	Cocoa tissue and arrowroot.		
79744	Baker's.....	4.85	21.65	8.96	1.97	6.99	78.0	10.93	6.21	"	"		
79745	Cowan's.....	4.25	26.15	7.54	5.03	2.51	33.0	6.21	"	"	"		
79746	Fry's U.S.A.....	4.06	25.20	7.56	4.81	3.27	32.0	5.36	"	"	"		
79747	Hershey's.....	3.35	21.45	8.16	3.00	6.16	64.0	8.64	"	"	"		
79748	Fry's Pure Un-sweetened.....	5.50	12.75	8.50	4.42	2.77	39.0	5.70	"	"	"		
79749	Lowney's.....	4.15	21.65	7.54	5.18	2.36	62.0	7.63	"	"	"		
79750	Cadbury's.....	5.30	22.65	10.76	2.09	8.67	31.0	5.30	"	"	"		
79751	White Star.....	5.70	20.75	9.86	2.35	7.61	77.0	8.90	"	"	"		
79752	Dr. Tibbles' Vi-Cocoa.....	4.55	23.85	10.14	2.31	7.83	77.0	9.51	"	"	"		